



## PRODUCTION OF BIOLUBRICANT BLEND FROM JATROPHA CURCAS OIL

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### ABSTRACT

Biolubricants derived from vegetable oils are environmentally compatible products due to their low toxicity and good biodegradability. Synthetic esters based on polyols and fatty acids possess suitable properties for lubricant applications, even at extreme temperatures. The current work investigates the oligomerization of fatty acid esters into biolubricant. The jatropha oil was esterified using recinoleic acid and isobutyric acid over activated carbon from kaolin as a catalyst to produce biolubricant. Also, the carbon was prepared from kaolin through impregnation and activated with copper nitrate solution. The generated activated carbon was characterized using FTIR and XRF. The biolubricant (triesters) were produced via oligomeric fatty acid esters with recinoleic acid under reflux at different reaction conditions and characterized using FTIR and GC-MS. Optimization of process parameters for oligomerization of jatropha oil with response surface based on Box-Beinkhen design. The process variables gave 90 min, 60°C and 1%, values corresponding to reaction time, reaction temperature and catalyst concentration as the optimal condition. A validation experiment was conducted to compare the optimal predicted value (93.07%) and experimental validated value (81.19%). The physicochemical properties of the jatropha oil and biolubricant produced were analyzed using ASTM methods. The quality parameters like kinematic viscosity, pour point, flash point viscosity index and blends of oil with that of commercial mineral oil were all in conformity with ASTM standard for biolubricant. The formulated oils also show the ability to significantly improve the kinematic viscosity, cold flow properties and possible potential as the replacement for the mineral-based lubricating oil.

**Keywords:** Biolubricants, Synthetic esters, jatropha oil

### INTRODUCTION

The current global energy originates from fossil sources, which would soon be exhausted in a short time due to their limited amount for a significant number of people. Besides, the growing use of fossil fuels at an increased rate will cause an escalation of local air pollution and exacerbate issues about the warming of the Earth as a result of CO<sub>2</sub> emissions (Shaldar *et al.*, 2019). New and sustainable lubricants are also needed to replace conventional lubricants derived from fossil fuels. This could solve many recent environmentally-related challenges and will resolve issues concerning their sustainability. (Hussein *et al.*, 2021; Nogales *et al.*, 2021) With additional problems surrounding climate change on a global scale, and exhaustion of oil reserves rising, the need for several environmentally friendly and sustainable options compared with current practices continues to gain maximum focus and attention (Sukirno *et al.*, 2020).

Over the last century, societies have strongly depended on fossil fuels, causing a progressive depletion of fuel reserves in such a way that it has been predicted that this non-renewable energy source will be exhausted in the medium term, thereby increasing search and development efforts for alternative chemicals and energy sources which could replace traditional fossil fuels. Many types of renewable energy sources, such as hydropower, geothermal, wind, solar, or biomass energy, have been proposed as potential energy sources. Among them, biomass is the only renewable source from which energy and chemical products can be obtained; therefore, this is the only current alternative able to replace petroleum in the synthesis of a wide range of valuable organic products.

It was reported that the annual production of lubricants is 30-40 million tons; these compounds are often used for many industrial applications to decrease friction and heat, protect against corrosion and wear, transmit energy, and eliminate

contaminants or sealing processes, among others (Saidur *et al.*, 2011; Menezes *et al.*, 2013) It was reported that about 50%–75% of total lubricant production is poured uncontrollably into the environment (Saidur *et al.*, 2011). About 95% of total lubricant production is petroleum-based, generally denoted as mineral oil. These oils are composed of a complex mixture of paraffinic (linear/branch), olefinic, naphthenic, and aromatic hydrocarbons of 20 to 50 carbon atoms. These formulations are non-renewable and toxic in such a way that the lubricants are harmful to humans and the environment as a consequence of their low biodegradability and high toxicity; some are even considered to be carcinogenic. However, biolubricants display faster and easier biodegradability, thereby exerting fewer adverse effects on the environment (Schneider *et al.*, 2006). Despite of the high toxicity of the biolubricants, lubricant discharge was also reported, causing strong contamination in air, soil, and drinking water. Considering the annual volume of lubricants and their environmental effects, governments have introduced strict regulations to mitigate the effects of the disposal of these lubricants (Erhan *et al.*, 2000).

### MATERIALS AND METHODS

#### Materials

The materials and reagents that will be used in carrying out this research are as follows: Crude Jatropha oil, Ethanol (99.1%), Potassium hydroxide (85.0%), Sodium hydroxide, Hydrochloric acid (36.0%), Sulphuric acid (98.6%), and phenolphthalein indicator. The instruments and equipment that will be used also in carrying out this research work are Soxhlet apparatus, Heating mantle, water bath, refrigerator, analytical balance capable of determining weights to four decimal places, conical flasks, Graduated cylinders, Beaker (difference sizes), three necks round bottom flasks, magnetic stirrer, retort stand and clamps, Liebig condensers,

Thermometer capable of measuring both negative and positive temperatures, pipettes and burette, mechanical stirrer and test tubes.

## Methods

### Catalyst Preparation

About 30 g of the Kankara white powdered kaolin will be weighed and soaked in (500 cm<sup>3</sup>) of distilled water into a pre-cleaned beaker (1000 cm<sup>3</sup>), the mixture will be ultrasonicated for 1h after which the suspended impurities will be decanted. The procedure will be repeated to ensure that the impurities are removed from the powdered clay and dried in an oven at 120°C for 2h (Bawa, 2012).

### Activation of the Kaolin

Exactly 25 g of the purified powdered kaolin will be soaked in 15 g copper nitrate solution (100cm<sup>3</sup>), the mixture will be subjected to constant stirring on a heating mantle with a magnetic stirrer (200rpm). The excess aqueous solution was dried using an oven. The copper nitrate on kaolin support was calcinated at 400°C for 4 h in a furnace to obtain the CuO on the support and the catalyst was stored in a polythene bag for analysis (Luna *et al.*, 2015; Ghulam *et al.*, 2013).

### Catalyst Characterization using FTIR and XRF

The surface functional group of the activated carbon was determined by using Fourier transform infrared (FT-IR) spectroscopic analysis (Carry630 Model Spectrophotometer). The analysis was performed by mixing dry carbon samples with potassium bromide (KBr) in a 1:20 weight ratio and ground into fine powder. The mixture was dried at 100°C for 3hrs, and thin pellets were made in manual equipment. The spectra were acquired at this temperature by accumulating 100 scans at 4-4000cm<sup>-1</sup>.

### X-ray Fluorescence (XRF)

Oxford instrument gave the elemental composition of the calcined catalyst. The atoms of the sample absorb energy from x-rays and become temporarily excited and then emit secondary x-rays. By measuring the intensity and characteristic energy of the emitted X-rays, qualitative and quantitative analysis regarding the thickness and composition can be obtained.

### Production of Biolubricant

Estolide ester will be synthesized in a one –step reaction in a 250 cm<sup>3</sup>, three –neck round bottom flask with a magnetic stirrer. Concentrated hydrogen peroxide will be used. And the heating mantle will be used to maintain the temperature of the reaction constant at 60°C ± 3°C. Recinoleic acid (50g, 0.1678mol) and branched fatty acid (0.083g/mol) will be combined and heated for 60°C temperature. Once the temperature reached 60°C, CuO catalyst (5%) will be added into the reaction and the flask will be placed under a vacuum and stirred for 6h, ethanol (34 cm<sup>3</sup>, 0.7391 mol) will be added into the flask and the reaction will continue for 3h in vacuum. As the reaction complete, it will neutralize with KOH (10 cm<sup>3</sup>, 2 mol) in 90% ethanol. The round bottom flask will be disconnected from the heating mantle and the solution will be left to cool down to room temperature. The particulate materials will be filtered through a Buckner funnel using whatman 2-filter paper, the organic layer will be transferred to a separation funnel and will be washed with distilled water to remove any excess ethanol employed for neutralization. The organic layer will be distilled using a simple distillation method at 80-140°C to remove any lactone by-products and excess ethanol and water (Abdalla *et al.*, 2018).

$$\text{Yield\%} = \frac{\text{Weight of Biolubricant}}{\text{Weight of oil}} \times 100$$

### Preparation of Blend Lubricant

Jatropha oil was mixed with mineral oil (lubricant SAE40) in various percentages from 10% to 50% of Jatropha oil instead of 10%. Mineral oil (lubricant SAE 40) will be used as baseline lubricant. The additive (CuO) will be added to the sample mixed and will be heated on the hot plate with a magnetic stirrer (200rpm) to obtain a homogenous mixture. The product obtained will be subjected to GC-MS or NMR to assess its chemical composition which will determine its lubricity and effective performance (Omotoso, & Akinsanoye, 2015).

### Data Analysis

The Biolubricant product yield obtained from the experiments conducted was analyzed on MINITAB 17 Statistical Software based on Box Beikhen design to estimate the main interaction effects on the reaction variables (i.e.; temperature, time, and catalyst loading), on the biolubricant product yield. The percentage product yield was fitted with a full quadratic polynomial model using regression analysis. The fitness of the model was evaluated by the coefficient of determination (R<sup>2</sup>) and the effects of terms were evaluated using analysis of variance (ANOVA) at a 95% confidence level. Contour plots were developed using the fitted quadratic polynomial equation. An optimizer on the MINITAB 17 was used to optimize the factors and the optimal level obtained was experimentally validated (Muhammad *et al.*, 2016).

### Characterization of Biolubricant product obtained

The Fourier transforms infrared (FT-IR) spectroscopic analysis and GC-MS analysis were carried out to determine the functional groups and chemical compositions of the biolubricant product.

### Physicochemical Properties of Jatropha Oil Biolubricant

#### Density and specific gravity determination

An empty beaker was weighed and recorded, and 50 cm<sup>3</sup> of the sample (Jatropha oil) was poured into the beaker and weighed. From the sample weight obtained, the density was determined by taking the ratio of the weight of the oil to the known volume (50 cm<sup>3</sup>) in SI units, according to the equations 1&2.

$$\text{Specific gravity} = \frac{\text{Weight of sample of oil}}{\text{volume of the oil}} \quad (1)$$

$$\text{Density} = \frac{\text{Weight of the oil}}{\text{Volume of water}} \quad (2)$$

#### Viscosity

The viscosity of the oil sample was determined at temperatures of 40 and 100°C, as follows, the sample was transferred to a 250 cm<sup>3</sup> beaker, and the temperature of the oil sample was raised to the desired value by heating on a heating mantle with constant stirring. The spindle was attached to the upper coupling by holding the coupling between the thumb and forefinger while carefully rotating the spindle counterclockwise. The spindle was immersed into the sample up to the middle of the indentation in the shaft. The viscometer was then turned on and allowed to run until a constant reading was attained.

#### Saponification value

The AOCS method Cd 3-25 was employed. 2 g of the sample (Jatropha oil) was weighed and transferred into a conical flask and 50 cm<sup>3</sup> of 0.5 M alcoholic KOH (that has stayed

overnight) was added to the sample. The mixture was then heated to saponify the oil. The unreacted KOH was back-titrated with 0.5 M hydrochloric acid using 2 to 3 drops of phenolphthalein indicator (Bilal et al., 2013). The saponification value (SAP) of the samples was analyzed and calculated using equation (3).

$$SAP\ value = \frac{(Titre\ value)(Molarity\ of\ NaOH)(56.1)}{(Weight\ of\ sample)} \quad (3)$$

**Acid Value**

Exactly 2 g of the sample (Jatropha oil) was weighed and transferred into a conical flask. The weight was recorded. 20 cm<sup>3</sup> of ethanol and 2-3 drops of the (phenolphthalein) indicator solution was added. And the solution was titrated against 0.5 M potassium hydroxide (KOH) solution with constant stirring until a faint, pink end-point appears. The endpoint value was then recorded, and the acid value was evaluated using equation 4) (Bilal et al., 2013).

$$Acid\ Value = \frac{(Titre\ value)(Molarity\ of\ KOH)(56.1)}{(Sample\ Weight)} \times 100 \quad (4)$$

**Iodine Value**

Exactly 0.5M was weighed into a 500 cm<sup>3</sup> glass-stopper flask with 10 cm<sup>3</sup> chloroform (CCl<sub>4</sub>). 10 cm<sup>3</sup> Hannus solution was measured into the flask and allowed to stand in a dark place for 30 minutes with occasional shaking. After incubation in the dark, 10 cm<sup>3</sup> potassium iodide solution was measured and added to the solution which was shaken thoroughly. 50 cm<sup>3</sup> of distilled water was added to wash down any free iodine on the stopper. A few drops of starch indicator were added and titrated against 0.1M sodium thiosulphate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>) with vigorous shaking so that any iodine remaining in the chloroform was taken up by the KI solution. Blank was also carried out in the same way but without a sample, and the volume of titrant was recorded using equation (5).

$$Iodine\ value = \frac{(Titre\ value) \times 0.01269}{Weight\ of\ sample} \times 100 \quad (5)$$

**Refractive Index**

The Abbey’s refractometer was reset with water. The oil was smeared on the lower prism of the instrument and closed. The light was allowed to pass by means of angle mirror and light appeared in the form of a dark band. The telescope tube was adjusted using fine until a black band appears in the cross-wire indicator, and it was recorded as a refractive index using equation (6).

$$Refractive\ Index = \frac{sinr}{siri} = 1.463 \quad (6)$$

**Preparation of Blend Lubricant**

Jatropha oil was mixed with mineral oil (lubricant SAE40) in various percentages from 10%, 20%, 30%, 40% and 50% of Jatropha oil instep of 10% of Mineral oil (lubricant SAE 40) was used as baseline lubricant. The samples were mixed and then heated on a hot plate with a magnetic stirrer (200 rpm) to obtained homogenous mixture. The product obtained was characterized and compared with standard to assess its lubricity and effective performance in an engine (Omotoso, and Akinsanoye, 2015).

**RESULTS AND DISCUSSION**

**Catalyst Characterization Using FTIR**

The IR spectra reveal the presence of metal-oxygen band in the region of 530-700 cm<sup>-1</sup>, attributed to the vibration of Cu-O and Al-O bonds, hence, confirming the presence of CuO and Al<sub>2</sub>O<sub>3</sub>. A report by Luna et al. (2015) confirmed the vibration modes in the range of 530-700 cm<sup>-1</sup> associated with CuO-nanoparticles. Abd El-Aziz et al. (2014) also observed a broad band in the range of 480-580 cm<sup>-1</sup> corresponding to CuO bond in its monoclinic structure. Saeid and Ali (2016) observed a strong band at 537 cm<sup>-1</sup> attributed to the vibration of Al-O confirming the formation of Al<sub>2</sub>O<sub>3</sub>. It can thus be concluded that the metal-oxygen frequencies observed for CuO and Al<sub>2</sub>O<sub>3</sub> are in close agreement with those of the literature values.

The chemical composition of the calcinated kaolin-supported activated copper oxide as a catalyst in Table 3.1 revealed that Copper Oxide (CuO) was the major component with a concentration of 42.66% as a result of calcination of the catalyst. The second and third major components are silicon oxide (SiO<sub>2</sub>) and aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) with a concentration of 39.63 and 34.54% respectively. In addition, small amounts of K<sub>2</sub>O, CaO, Fe<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub>, ZnO, PbO, and MnO were also observed as the impurities present in the sample. XRF was conducted by Ibrahim et al., (2023) on the metakaolin based alkali-activated materials (AAMs) at 800°C but the amount of CuO obtained was 56.80% which was the higher compared to the amount of CuO oxide obtained in this research 42.66%. The low or minimum amount of CuO obtained from this work can be due to higher operational temperature used upon calcination at 400°C, also the coordination of the copper nitrate with kaolin ratio may result in low copper oxide catalyst. This reveals that CuO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> are the major oxides on the calcinated kaolin catalyst.

**Table 1: XRF Analysis Result of the Catalyst**

Compound	Concentration (wt %)
Al <sub>2</sub> O <sub>3</sub>	34.54
SiO <sub>2</sub>	39.63
K <sub>2</sub> O	1.140
CaO	0.096
Cr <sub>2</sub> O <sub>3</sub>	0.023
Fe <sub>2</sub> O <sub>3</sub>	0.543
<b>CuO</b>	<b>42.66</b>
ZnO	0.002
PbO	0.008
MnO	0.012

**Effect of the Process Variables on the Yield of Biolubricant**

The analysis of variance (ANOVA). Presented in Table 4.6 shows the effect of the production variables (reaction temperature, reaction time and catalyst loading). The ‘p’

values were used to identify whether the variables or their interactions are statistically significant or not. The variables with p-values greater than alpha value (α=0.05) is identified to be statistically insignificant and vice versa. The correlation coefficient R-square (R<sup>2</sup>) of the model is 87.3 %, which

indicates that the variables fit the models. The variables constant, reaction temperature, reaction time\*reaction time, catalyst loading\*catalyst loading, and reaction time\*catalyst loading were statistically significant on the biolubricant yield. The variables catalyst loading, reaction time, reaction temperature\*reaction temperature, reaction time\*reaction temperature and reaction temperature\*catalyst loading have p-values greater than  $\alpha$ -value which shows that they are statistically insignificant on the biolubricant yield.

**Effect of Reaction Temperature on the Yield of Biolubricant**

The contour plot presented in Figure 4.2 shows the effect of reaction temperature on biolubricant yield. As the temperature increases from 50 to 70°C, the biolubricant yield significantly

increases. Initially, some thermal energy was needed for estolide formation as the reaction was endothermic (Samart *et al.*, 2009). Since the reaction mixture constitutes a different phase system, the thermal energy was sufficiently needed to overcome the diffusion resistance between the phases. However, the high temperature is not preferred, as the temperature increases and reaches the boiling point they vaporize immediately and form a large number of bubbles, which inhibits the reaction and decreases the biolubricant yield (Salaheldeen, *et al.*, 2021).

Additionally, Figure 4.2 shows the effect of the reaction between time and temperature against Biolubricant yield the response surface of the extent of conversion showed a clear peak, suggesting that the optimum condition for maximum yield is within the design boundary.

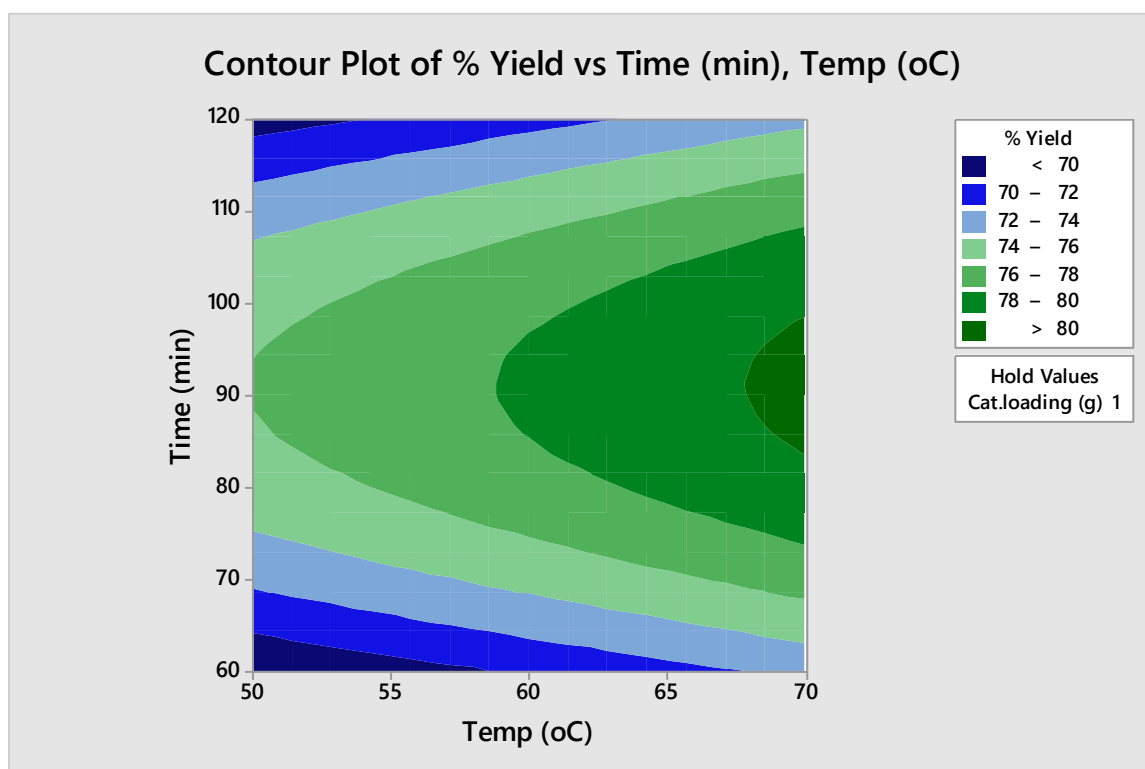


Figure 1: Effect of reaction temperature on biolubricant yield.

**Effect of Catalyst Loading on the Yield of Biolubricant**

Figure 4.3 shows the decrease in the biolubricant yield with increases in the catalyst loading. The optimum catalyst loading was found to be 1 g with a jatropha biolubricant yield of 78.40%. The excess catalyst concentration increases the viscosity of the reactant which also lowers biolubricant yield as reported by (Salaheldeen, *et al.*, 2021). Figure 4.3 shows

the effect of the interaction of catalyst loading with reaction time on biolubricant yield which indicates that as the reaction time increases from 1.0 to 2h at 1 w/w% catalyst loading, the biolubricant yield increases but as the catalyst loading goes beyond 1 w/w% the yield decreases which is to say the little amount of catalyst loading is needed and thereby save production cost.

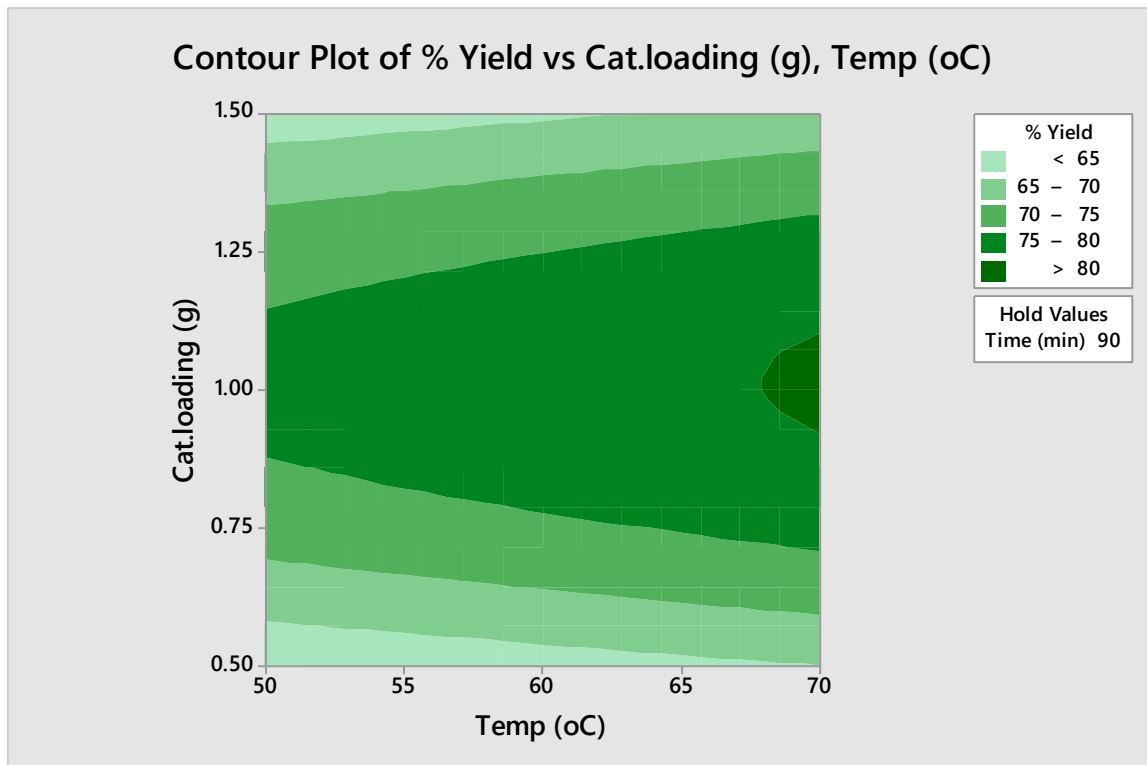


Figure 2: Effect of interaction of catalyst loading and reaction temperature on biolubricant yield

**Effect of Reaction Time on Biolubricant Yield**

Figure 4.4 shows the effect of reaction time on biolubricant yield. As the reaction time increases from 1-2hrs, the

biolubricant yield rapidly increases until the reaction has reached the equilibrium, beyond the optimum yield reaction time (2.5hr), a reverse reaction occurs (Adeyemi, *et al.*, 2020).

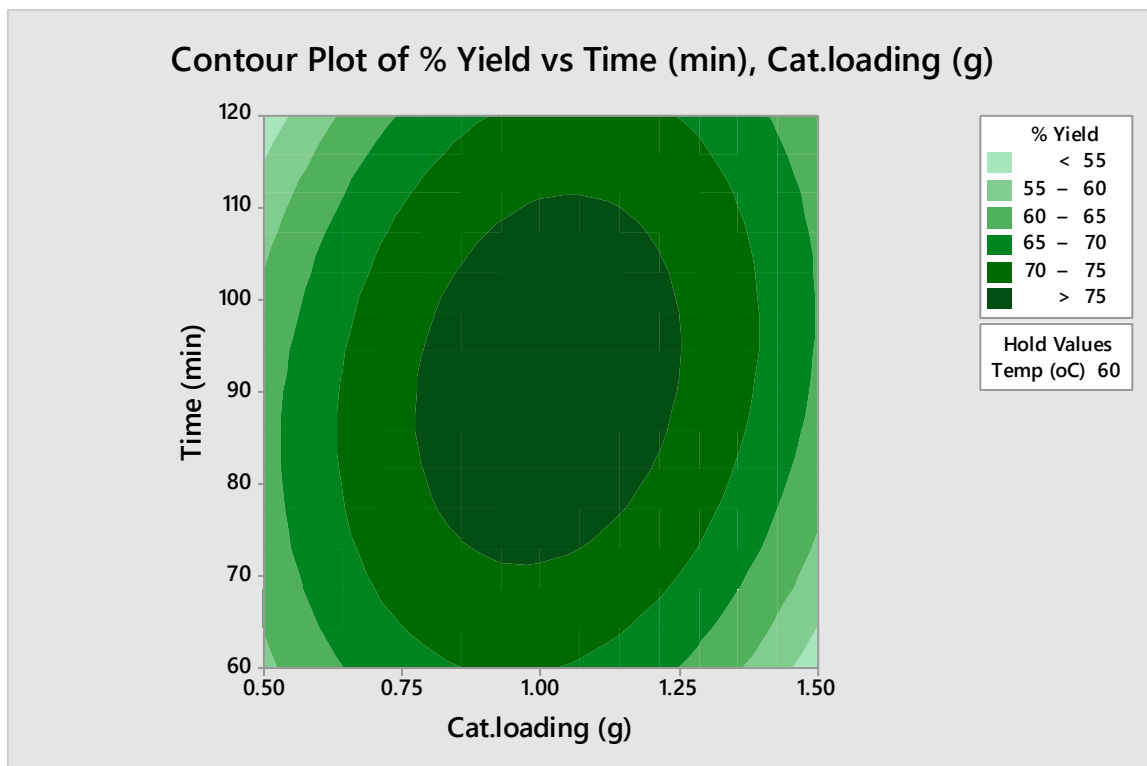


Figure.3: Effect of interaction of catalyst load and reaction time on biolubricant on yield

**Response Variables and Validation**

Table 2 shows the predicted results for the optimal solution obtained from the optimization. The solutions predicted

maximum biolubricant yield of 80.48 and 76.24% and desirability of 1.00000 and 0.91365 for the two solutions with optimal process conditions of temperature (70 and

69.6°C), time (69 and 92 min) and catalyst loading of (1.01 and 0.95 wt%) for the two solutions respectively.

Biolubricant yield of 80.48 and 76.24 % was obtained from validation experiment carried out at the levels of the process variables predicted in solution 1 and 2. It was observed that the optimized predicted and experimental validated results for the two solutions were 80.48%, 76.24 % and 81.19 %, 76.10

%. Thus, with predicted yields of 80.48 % and 76.24 % which is closer to the experimental yields of 81.19 % and 76.10 %. Therefore, it can be concluded that the optimization model is reliable and may therefore be more attractive for larger scale biolubricant production from estolide formation process catalyzed by kaolin-supported activated copper oxide as catalyst. The optimization plot is presented in Figure 3

**Table 2: Predicted Result of Optimization and Validation of Biolubricant Yield**

Solution	Temperature (o C)	Reaction Time (min)	Cat. Load (g)	Yield		Desirability
				Predicted	Experimental	
1	70	91.52	1.01	80.48	81.19	1.00000
2	69.62	60	0.95	76.24	76.10	0.91365

**Result for Physicochemical Properties of Biolubricant**  
**Result of Kinematic Viscosity of the Biolubricant at 40 & 100°C**

The most important physical properties of lubricating oil are the kinematic viscosity (Ratchadaporn *et al.*, 2015). Viscosity is very important in determining the fluidity of lubricating oil at low and high temperature (Bilal *et al.*, 2013).

**Result of Viscosity Index of the Biolubricants**

The viscosity index (VI) is an arbitrary number indicating the effect of changing temperature on the kinematic viscosity of lubricating oil (Ratchadaporn *et al.*, 2015).

**Result of Flash Point of the Biolubricant**

The minimum temperature at which lubricating oil generates flammable vapours which can be ignited in the air by a flame about it surface is called the flash point of the oil (Janes and Chaineaux, 2013).

**Result of Pour Pour Point of the Biolubricant**

Poor low-temperature property is a limiting factor in large-scale applications of triglycerides oils-based lubricants (Weimin and Xiaobo, 2015; and Musa *et al.*, 2015). Triglycerides oils tends to form macro crystalline structures at low temperatures via uniform stacking of triglycerides' backbones and such macro crystals limit the easy flow of the fluids due to the loss of kinetic energy of the molecules during this self-stacking (Weimin and Xiaobo, 2015).

**Result of Acid Value of the Biolubricant**

The concentration of acidic compounds in lubricating oil can lead to the corrosion of machine parts and clogged filters due to the formation of varnishes and sludges. Oils with high acid values (AV) are susceptible to decomposition (Omahu and Omale, 2017).

**Result of Iodine Value of the Biolubricant**

The iodine value is a measure of the amount of double bonds present in the molecules of a given sample of oil (Adolf *et al.*, 2018).

**Table 3: Result for Physicochemical Properties of Biolubricant**

Parameters	KV@100°C (cSt)	KV@40°C (cSt)	VI	FP (°C)	PP (°C)	AV (mg/KOH)	IV
(mgI <sub>2</sub> /g)							
<b>JSO</b>	9.96±0.03	54.52±0.04	131±0.00	214±1.10	-10.3±0.02	5.87	29.78
<b>Biolube</b>							
<b>ASTM</b>	-	>41.4	>90	220	< -10	-	-

**GC-MS Profile of the Biolubricant Produced**

The Biolubricant was produced from jatropha seed oil using kaolin supported activated copper oxide as a catalyst. The reaction was carried out in a 250 cm<sup>3</sup> round bottom flask and heated under reflux on a hot plate equipped with a magnetic stirrer. The product was also analyzed using GC-MS. The

results in Table 4 shows the distribution of the esters obtained from the estolide formation process using kaolin supported activated copper oxide as catalyst. Furthermore, the analysis of GC-MS of the products (Appendix iv) showed that other organic products detected by GC-MS are most likely the by-products of the reaction.

**Table 4: GC-MS Analysis of Biolubricant**

Compounds	Peak Area (%)
Hexamethyl-1,4-Bis(trimethylsilyl)benzene	0.38
Cyclotrisiloxane	
9,12-Octadecadienoic acid ethyl ester	5.43
Undec-10-ynoic acid, undecyl ester	1.98
Cis/trans-13-Octadecenoic acid	2.73
(Z)-9-Octadecenoic acid-2-hydroxy-1-(hydroxymethyl)ethyl ester	14.00
15-hydroxypentadecanoic acid	4.96
(Z)-9,17-Octadecadienal,	3.10
(Z, Z)-9,12-Otadecadien-1-ol,	
2-hydroxy-1- (hydroxymethyl)ethyl ester	14.60
6,11-dimethyl-2,6,10-dodecatrien-1-ol	0.22
Butyl-9-Octadecenoate	2.09

1,2-Benzisothiazole	2.63
9-Octadecenoic acid (Z)-, 2-hydroxy-1-(hydroxymethyl)ethyl ester	2.35
(Z)-9-Octadecenoic acid, 2,3-dihydroxypropyl ester	5.63
9-Tricosane	3.22
(Z)-6-Octadecenoic acid cyclodocosane	2.29

### The Result of Physicochemical Properties of Blended Oils Kinematic Viscosity of the Oil Blends with SAE 40

The measure of oil's internal resistance to its flow is viscosity (Ozioko, 2014; Dabai *et al.*, 2018). And is used in the identification of lubricating oil grades and monitoring the changes occurring in the oils while in service (Kalam *et al.*, 2012). Higher viscosity is an indication of deterioration by oxidation or contamination, while a decrease in viscosity suggests dilution by fuels (Kus, 2011; Dabai *et al.*, 2018). The viscosity of blended lubricants is presented in Table 4.10. It shows that the kinematic viscosity of Jatropha oil biolubricant increases with increasing blending ratio from 10.00-20.00 (wt) at both 40 and 100°C. This suggests that as more jatropha oil lubricant is being added to SAE 40 at 40 and 100°C, the viscosities of the blend will increase. In another view, Imran (2013) who worked on the blends of 10, 20, 30, 40 and 50% (wt) of jatropha bio lubricant with SAE 40 reported that the viscosity of JBL 10 and 20 (jatropha bio lubricant lubricating oil) satisfied SAE 40 grade requirements at both 40 and 100 °C and also met the ISO grade 100 standard. However, 30, 40 and 50% (wt) do not meet the requirement of ISO grade 100 at both temperatures (40 & 100). Therefore, the blended JBL has comparable viscosity with that of SAE 40 lubricating oil and can replace SAE 40 in any application.

### Viscosity Index of the Oil Blends with SAE 40

Viscosity index of any lubricating oil is inversely proportional to its temperature and therefore, a machine using such oil and operating over a higher temperature range requires a high viscosity index (Nehal and Amal, 2013; and Dabai *et al.*, 2018). Viscosity indices of the blended bio lubricants are presented in Table 4.10. the trend suggests that the higher the ratio of vegetable oils with SAE 40, the higher the Vis of the blended oils. The reason is that the control SAE 40 is viscous enough to change at a higher temperature range and adding and oily material makes its viscosity index to be much higher than what was used as a control. From the result obtained on the blended bio lubricants, it can be inferred that they have the potential to be used as a lubricant for a one-stroke engine.

### Flash Point of the Oil Blends with SAE 40

Volatility and fire resistance are important parameters to be considered in the transportation and storage of lubricating oils. Flash points (FP) are the lowest temperature at which vapourised lubricating oil can be ignited by an external light source. It is also the minimum temperature at which oil gives sufficient vapours which enables it to ignite momentarily when a flame of standard dimension is brought near the surface of its vapour for a prescribed rate in apparatus of specified dimension (Kailas *et al.*, 2015). The flash points of various blended lube oils are presented in Table 4.10. The results indicate that the more the quantity of vegetable oils

with SAE 40, the higher the flash points of the blends. The FPs of these blends are also within the values obtained by Danjuma and Dandago (2009), and Mukhtar *et al.* (2014) hence, are safe for storage and transportation.

### Pour Point of the Oil Blends with SAE 40

The pour point of an oil is an important parameter that should be considered for an operation in cold environment or equipment that handles cold fluid. At low temperature, the viscosity of oil sample will be very high and therefore, causing the oil to resist flow. The temperature at which oil just ceases to flow when cooled in a specified rate in standard apparatus is the pour point of that oil (Kailas *et al.*, 2015). The pour point of the blended vegetable biolubricants is presented in Table 4.10. The value decreases with increasing ratio of the vegetable oils in the blend. Arianti and Widayat (2018). State that vegetable oils with more double bonds in their chains will have lower pour points compared to those with fewer double bonds. The pour point of the blended oils is lower. Therefore, will course no effect in a very low temperature condition unlike the SAE 40.

### Acid Value of the Oil Blends with SAE 40

Acid value (AV) is a measure of the acid concentration present in a given sample of lubricating oil. The acid values of the biolubricant blends are presented in Table 4.10. The analysis shows that the higher the ratio of biolubricant in the blends, the higher the acid value of the blend. This was confirmed that vegetable oils contain a greater number of fatty acids and oxygen molecules in their chains than the mineral-based oils (Kalam *et al.*, 2012; Adolf *et al.*, 2018; Muhammad *et al.*, 2018). Generally, all the acid values of the blends agree with that reported by Danjuma and Dandago (2009), and Mukhtar *et al.* (2014). Who investigated blends of jatropha oil and suggested that the oil has the potential to be used as an engine oil lubricant.

### Iodine Value of the Oil Blends with SAE 40

Iodine value (IV) is a measure of the degree of unsaturation of the lubricating oil molecular structure (Gobinda *et al.*, 2017). The iodine values of the blends are presented in Table 4.10. It was found that all the iodine values of the blends increase as the quantity of the biolubricant increases, which suggests that the degree of unsaturation of the blends increase with increasing ratio and therefore, the more the quantity of biolubricant in the blends, the more susceptible the blends to oxidation reactions.

Vegetable oils with unsaturated fatty acids in large amount are susceptible to oxidation compared to those with fewer C=C bonds. When these oils are treated otherwise or the chain length is reduced, they behave differently and making them a better alternative to petroleum-based lubricants.

**Table 5: Physicochemical properties of Jatropha Seed Oil (JSO) Ranging from B10, B20, B30 B40 & B50 with Mineral Oil (SAE40).**

Blended Oil (mgI <sub>2</sub> /g)	KV@100° C (cSt)	KV@40° C (cSt)	VI	FP (°C)	PP	AV (°C)	IV (mgKOH/g)
JBL10 49.02	10.09±0.34	43.22±0.26	161.98	209±0.32	-	22	3.62
JBL20 58.13	12.26±0.38	46.13±0.19	170.00	213±0.87	-	18	3.81
JBL30 65.26	15.13±2.11	50.34±0.43	179.40	217±0.48	-	15	4.01
JBL40 75.08	16.45±0.41	54.34±0.41	186.98	226±0.21	-	11	4.13
JBL50 83.04	18.16±0.25	58.12±0.51	198.20	233±0.13	-	9	5.20

Key: Values are expressed in triplicates. KV= Kinematic Viscosity, VI- Viscosity Index, FP=Flash Point, PP= Pour Point, AV= Acid Value, IV= Iodine Value.

### CONCLUSION

This study was conducted to produce as ecofriendly biolubricant from jatropha seed oil, and the blend of the oil with SAE 40 lubricating oil. The following conclusions were deduced from the analyses carried out:

The jatropha oil was successfully extracted from Jatropha seed using a Soxhlet extractor. The physicochemical properties of the jatropha oil were conformed with ASTM standard. The catalyst produced (kaolin/copper nitrate) successfully and effectively converted the jatropha oil into biolubricant. The optimum yield of biolubricant (78.4%) was achieved at 90 min. reaction time, 60 °C temperature, 1.0 wt% catalyst loading. The physicochemical properties of the biolubricant produced show an improvement in kinematic viscosity, cold flow properties and thermal and oxidative stability of the biolubricant. The prepared blends of the mineral oil (SAE 40) with produced biolubricant (JBL10, JBL20, JBL30, JBL40 and JBL50) have significantly improved the properties of the mineral oil and were in conformity with ISO VG standards.

### REFERENCES

Abdalla, B. K. (2018). Biofuels, Bio Lubricants Production for Industrial Application, The Sudanese Experience, *Science Environmental*, **1**: 39-41.

Adolf, O., Akwasi, A., Gyang, N. O., Amoa, C. A., and Akofa, A. A. (2018). Comparative Assessment of Some Physico-chemical Properties of Seed Oils of Parkia biglobosa and Monodora myristica with some Commercial Oils. *African Journal of Food Science*, **12**:1-5.

Arianti, W. A. and Widayat, W. (2018). A Review of Biolubricant Production from Vegetable Oils Using Esterification Transesterification Process. *MATEC Web of Conference*, **156**:1-7.

Bawa, S. G. (2012). Development of Gamma Alumina from Kankara Kaolin for Potential Application as Catalysts Support. (M. Sc Thesis, Department of Chemical Engineering, Ahmadu Bello University, Zaria).

Dabai, M. U., Owuna, F. J., Sokoto, M. A. and Abubakar, A. L. (2018). Assessment of Quality Parameters of Ecofriendly Biolubricant from Waste Cooking Palm Oil. *Asian Journal of Applied Chemistry Research*, **1**:1-11.

Danjuma, M. N. and Dandago, M. A. (2009). Extraction and Characterization of Calabash (*Langeneria Siceratia*) Seed Oil. *Techno Science Africana Journal*, **3**:67-69.

Erhan, S. Z., & Asadauskas, S. (2000). Lubricant base stocks from vegetable oils. *Industrial Crops Production*, **11**, 277–282.

Ghulam, M., Hajira, T., Mohammed, S. & Nasir, A. (2013). Synthesis and characterization of Cupric Oxide (CuO) Nanaoparticles and their application for the removal of dyes. *African Journal of Biotechnology*. **12**; 6650-6660.

Gobinda, K., Pranab, G. and Bragendra, K. S. (2017). Chemically Modified Vegetable Oils to Prepare Green Lubricants. *Lubricants*, **44**:1-17.

Hussein, R. Z. K., Attia, N. K., Fouad, M. K., & ElSheltawy, S. T. (2021). Experimental investigation and process simulation of biolubricant production from waste cooking oil. *Biomass Bioenergy*, **144**,105850.

Kalam, M. A., Masjuki, H. H., Shahabuddin, M. and Mofijur, M. (2012). Tribological Characteristics of Amine Phosphate and Oxylated/Butylated Diphenylamine Additives Infused Biolubricant Energy Education Science and Technology Part A: *Energy Science and Research*, **30**: 123-136.

Luna, I. Z., Hilay, L. N., Chowdhung, A. M. S., Gafur, M. A., Khan, N. & Khan, A. A (2015). Preparation and characterization of Copper Oxide Nanoparticles synthesized via Chemical Precipitation Method. *Open Access Journal*, **2**:1-8.

Menezes, P. L., Ingole, S. P., Nosonovsky, M., Kailas, S. V., & Lovell, M. R. (2013). *Tribology for Scientists and Engineers*.

Musa, U., Mohammed, I. A., Sadiq, M. M., Aberuagba, F., Olunrude, A. O. and Obamina, R. (2015). Synthesis and Characterization of Trimethylpropane-Based Biolubricants from Castor Oil. Proceedings of the 45<sup>th</sup> Annual Conference of NSChE, 5<sup>th</sup> -7<sup>th</sup> Nov, 2015. Warri: Annual Conference of NSChE, 5<sup>th</sup> – 7<sup>th</sup> Nov, 2015: 248-253.

Omotoso, M. A., & Akinsanoye, O. A., (2015), A review of biodiesel generation from non-edible seed oils using non-conventional heterogenous catalysts, *Journal of Petroleum Technology and Alternative fuels*, **6**:1- 12.



Saidur, R., Abdelaziz, E. A., Demirbas, A., Hossain, M. S., & Mekhilef, S. (2011). A review on biomass as a fuel for boilers. *Renewables Sustainable energy revolution*, **15**:2262–2289.

Salaheldeen, M., Mariod, A. A., Aroua, M. K., Rahman, S. M. A., Soudagar, M. E. M., and Fattah, I. M. R. (2021). Current state and perspectives on transesterification of triglycerides for biodiesel production. *Catalysts*, **11**: 1–37.

Schneider, M. P. (2006). Plant-oil-based lubricants and hydraulic fluids. *Journal of science food and agriculture*, 1769–1780.

Shaldar S. P., Athith, D., & Abhishek, H. A., (2019). Biodiesel production, optimization and fuel properties characterization of waste fish oil. *International Resolution Journal of Engineering Technology*, **6**:947-952.

Sukirno, S., Triana, A. S., & Rifqi, M. (2020). Biolubricant from spent bleaching earth oil through propylene glycol Ester synthesis. *International Tropical Renewable Energy Conference*, 2255.



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